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PATENT
CASE 4233C3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Donald B. Appleby et al.	:	Group Art Unit: 1211
Serial No.:	08/360,184	:	Examiner: E. White
Filed:	December 20, 1994	:	
For:	**Polyol Polyester Synthesis**		

DECLARATION UNDER 37 C.F.R. §1.608 OF STEVEN R. ALEXANDER

Assistant Commissioner for Patents
Washington, DC 20231

Dear Sir:

I, STEVEN R. ALEXANDER, declare that:

1. I am employed by the assignee of the present application, The Procter & Gamble Company, and have been working for The Procter & Gamble Company continuously since 1983.

2. From 1988 to the present I worked first as a technician and later as a scientist on the sucrose polyester synthesis project, and I was responsible for conducting laboratory experiments relating to the synthesis of polyol fatty acid esters, specifically sucrose fatty acid esters, commonly referred to as sucrose polyesters, FG or FG base, by the reaction of sucrose and fatty acid methyl

esters, according to predetermined test plans and analyzing the results according to established procedures.

3. I conducted and analyzed the results of a variety of sucrose polyester laboratory experiments during 1989 and 1990 under the direction and control of Mr. Ju-Nan Kao and/or Mr. Scott D. Pearson, including those described in this Declaration.

4. For each experiment which I conducted and which is described in this Declaration, I accurately recorded, on or about the day of the experiment, the general nature of the experiment, including pertinent reaction parameters, the results of the experiment, the date of the experiment and my signature in a Laboratory Notebook provided to me for such a purpose.

5. I have examined Exhibit 46 and I confirm that Exhibit 46 comprises accurate copies of pages 40-41 and 44-46 of Laboratory Notebook SI 1386 on which I accurately recorded a series of experiments I conducted in September and October of 1989, and which I signed and accurately dated upon the completion of the procedures described on each page respectively. These laboratory experiments were conducted according to the instructions set forth by Ju-Nan Kao on page 39 of Laboratory Notebook SI 1386, Exhibit 52, as discussed in the Kao Declaration, ¶14. Laboratory Notebook SI 1386 was in my possession and control from September 1989 through June 1990.

6. Pages 40-41 of Exhibit 46 describe a sucrose polyester reaction which I conducted on September 27, 1989 and which was part of several experiments intended to evaluate, inter alia, the effect of filtration during the sucrose polyester reaction, as described in Exhibit 52, page 39, line 4. As described at page 40, lines 22-31 and page 41, lines 1-4 of Exhibit 46, the reaction mixture

in reactor R303 was filtered during the reaction by passing the reaction mixture through a 5 micron filter. This filtration removed soap, unreacted or burned sucrose and carbonate solids from the reaction mixture. As set forth on page 41, lines 1-6, the filtered reaction mixture was placed in reactor R304 and the reaction was continued. The reaction mixture was sampled before and after the filtration, as noted at page 41, line 3.

7. Lines 19-32 of page 41 set forth the average degree of esterification or I-bar ("$\bar{I}$") of the sucrose ester product, the percent unreacted sucrose ("% Sucrose"), the percent of total sucrose esters as octaester ("% Octa") and the percent of soap ("% Soap") in the reaction mixture as a function of time. As set forth at lines 23-24 of page 41, the sucrose ester product in the reaction mixture before filtering had an I-bar of about 4.6 (which corresponds to an average degree of esterification of 57%), while the filtered reaction mixture after filtering comprised about 0.05% unreacted sucrose. As further indicated, sucrose ester product in the reaction mixture contained about 99% octaester about 2 hours after the filtration.

8. Pages 44-46 of Exhibit 46 describe a sucrose polyester reaction which I conducted on October 2, 1989 and which was part of several experiments intended to evaluate, inter alia, the effect of filtration during the sucrose polyester reaction. As described at page 45, lines 7-12, the reaction mixture in reactor R303 was filtered during the reaction using a 1 micron filter. A control reaction was performed in reactor R306 wherein the same reactants used in the initial stage of reactor R303 were employed as described at page 44, lines 30-32. As noted at lines 7-12 of page 45, the initial stage reaction mixture in reactor R303 was passed through the 1 micron filter and then placed in reactor R304. As noted at page 45, lines 13-14, a portion of the unfiltered reaction mixture from reactor R306 was transferred to reactor R303. The reaction was then continued in each of

reactors R303 (unfiltered), R304 (filtered) and R306 (unfiltered). Reactor R303 was provided with nitrogen sparging by a diffusion stone which produced fine bubbles, while Reactors 304 and 306 were provided with nitrogen sparging by a tube which produced large bubbles.

9. Lines 14-21 of page 46 set forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("Octa") in the reaction mixture for reactors R303 (unfiltered, fine bubble sparging), R304 (filtered, large bubble sparging) and R306 (unfiltered, large bubble sparging) as a function of time subsequent to the time of completion of the filtration step. As indicated, the sucrose ester product in the filtered reaction mixture of reactor R304 comprised about 98% octaester about 2 hours after the filtration while the sucrose ester products in the unfiltered reaction mixtures in reactor R303 and reactor R306 each comprised about 77% octaester at about that same time.

10. I have examined Exhibit 47 and I confirm that Exhibit 47 comprises accurate copies of pages 105-107, 111-112 and 114-115 of Laboratory Notebook SI 1386 on which I accurately recorded a series of experiments I conducted in December 1989 and January 1990, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

11. Pages 105-107 of Exhibit 47 describe a sucrose polyester reaction which I conducted on December 12, 1989 and which was one of several experiments intended to evaluate, inter alia, the effects of reaction temperature and filtration during the sucrose polyester reaction. As described at page 106, line 10, about one half of the reaction mixture in reactor R303 was filtered during the

reaction and the filtrate was transferred to reactor R304. The reaction was then continued in each of reactors R304 (filtered) and R303 (remaining unfiltered) at a temperature of about 295°-300° F.

12. Lines 1-23 of page 107 of Exhibit 47 set forth the I-bar, ("IBAR") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") in the reaction mixture for reactors R303 (unfiltered) and R304 (filtered) as a function of time subsequent to completion of the filtration step. Prior to filtration, the sucrose ester product in the reaction mixture in reactor R303 had an I-bar of about 2.91 (which corresponds to an average degree of esterification of about 36%), as described at page 107, line 3. Shortly after filtration, the filtered reaction mixture in reactor R304 comprised about 0.19 weight percent unreacted sucrose, as described at page 107, line 16. As indicated, the sucrose ester product in the filtered reaction mixture in reactor R304 comprised about 70.9% octaester and had an I-bar of about 7.61 about 2 hours after the filtration while the sucrose ester product in the unfiltered reaction mixture of reactor R303 comprised about 44.6% octaester and had an I-bar of about 7.15 at that same time.

13. Pages 111-112 of Exhibit 47 of Exhibit 47 describe a sucrose polyester reaction which I conducted on January 10, 1990 and which was one of several experiments intended to evaluate, inter alia, the effect of temperature and filtration during the sucrose polyester reaction. As described at page 111, lines 17-18, about one half of the reaction mixture in reactor R303 was filtered through a 1 micron filter during the reaction and the filtrate was transferred to reactor R304. The reaction was then continued in each of reactors R304 (filtered) and R303 (remaining unfiltered). As described at page 111, line 21-page 112, line 7, the temperature of the reaction in each reactor after filtration was about 250°F.

14. Lines 10-31 of page 112 of Exhibit 47 set forth the I-bar ("I-BAR") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") in the reaction mixture in reactors R303 (unfiltered) and R304 (filtered) as a function of time subsequent to completion of the filtration step. Prior to filtration, the sucrose ester product in the reaction mixture in reactor R303 had an I-bar of about 1.95 (which corresponds to an average degree of esterification of about 24%), as described at page 112, line 20. The filtered reaction mixture of reactor R304 comprised about 0.71% unreacted sucrose after filtering, as described at page 112, line 23. As indicated, the sucrose ester product in the filtered reaction mixture in reactor R304 comprised about 11.8% octaester and had an I-bar of about 5.56 about 2 hours after the filtration while the sucrose ester product in the unfiltered reaction mixture of reactor R303 comprised about 5.1% octaester and had an I-bar of about 4.67 at that same time. Additionally, the sucrose ester product in the filtered reaction mixture in reactor R304 comprised about 79.3% octaester and had an I-bar of about 7.77 about 7 hours after the filtration while the sucrose ester product in the unfiltered reaction mixture of reactor R303 comprised about 44.0% octaester and had an average degree of esterification of about 7.09 at that same time. The column labeled "DFK" on page 112 sets forth difatty ketone levels in parts per million (ppm) and, as described on line 31, the DFK ketone level in the product of the filtered reaction mixture about 7 hours after filtration was about 165 ppm.

15. Pages 114-115 of Exhibit 47 describe a sucrose polyester reaction which I conducted on January 12, 1990 and which was one of several experiments intended to evaluate the effect of temperature and filtration during the sucrose polyester reaction. As described at page 114, lines 17-18, about one half of the reaction mixture in reactor R303 was filtered through a one micron filter during the reaction and the filtrate was transferred to reactor R304. The reaction was then continued

in each of reactors R304 (filtered) and R303 (remaining unfiltered). As described at page 114, line 20-page 115, line 2, the temperature of the reaction after filtration was about 295°-320°F.

16. Lines 8-26 of page 115 set forth the I-bar ("I-BAR") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") of the reaction mixture in reactors R303 (unfiltered) and R304 (filtered) as a function of time subsequent to the filtration step. Prior to filtration, the reaction mixture of reactor R303 had an average degree of esterification of about 1.90 (which corresponds to an average degree of esterification of about 24%), as described at page 115, line 10. The filtered reaction mixture in reactor R304 comprised about 0.73% unreacted sucrose as described at page 115, line 19. Additionally, the sucrose ester product in the filtered reaction mixture of reactor R304 comprised about 63.9% octaester and had an I-bar of about 7.51 about 2 hours after the filtration while the sucrose ester product in the unfiltered reaction mixture of reactor R303 comprised about 24.4% octaester and had an I-bar of about 6.59 at that same time. The sucrose ester product in the filtered reaction mixture in reactor R304 comprised about 79.8% octaester and had an I-bar of about 7.77 about 6 hours after the filtration while the sucrose ester product in the unfiltered reaction mixture of reactor R303 comprised about 35.0% octaester and had an I-bar of about 6.98 at that same time. The column labeled "DFK" on page 115 indicates that the filtered reaction mixture product contained about 320 ppm DFK about 6 hours after filtration.

17. I have examined Exhibit 48 and I confirm that Exhibit 48 comprises accurate copies of pages 118-139 of Laboratory Notebook SI 1386 on which I accurately recorded a series of experiments I conducted in April, May and June 1990, and which I signed and accurately dated upon the completion of the procedures described on each page respectively.

18. Pages 118-122 of Exhibit 48 describe three sucrose polyester reactions which I conducted on April 30 and May 1-2, 1990, and which were intended to evaluate, inter alia, the use of a packed column reactor during a later stage of the sucrose polyester reaction. As described at page 118, lines 1-9, a goal of these experiments was to achieve a sucrose polyester product comprising 75% octaester, by weight of total sucrose esters, using less vacuum (higher pressures), lower temperatures and lower ester to sucrose molar ratios in the column reactor. As set forth at page 120, lines 4-15, in the first experiment, the packed column reactor was operated at a pressure of about 15-33 mm Hg and a temperature of from about 243°-267°F. As set forth at page 121, line 12, in the second experiment, the packed column reactor was operated at a pressure of about 50 mm Hg and a temperature of about 275°F. As set forth on page 122, line 16, in the third experiment, the packed column reactor was operated at a pressure of about 5 mm Hg and a temperature of about 275°F. Pages 120-122 set forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") in the reaction mixture as a function of time in the column for each of the three experiments, respectively.

19. Pages 123-124 of Exhibit 48 describe a sucrose polyester reaction which I conducted on May 4, 1990 and which was intended to further evaluate the use of the packed column reactor during a later stage of the sucrose polyester reaction. As described at page 123, line 29-page 124, line 7, the top of the packed column reactor was operated at a pressure of from about 50 to about 51 mm Hg and a temperature of from about 265°F to about 280°F (from about 130°C to about 138°C). The middle of the column was operated at a temperature of from about 125°C to about 110°C, while the bottom of the column was operated at a temperature of from about 109°C to about 81°C and a pressure of from about 68 to about 72 mm Hg. As described at page 123, line 16, initially the molar

ratio of ester to sucrose was 8.5:1, corresponding to a molar ratio of ester to esterifiable sites on the polyol of 1.06:1.

20. Lines 10-17 of page 124 set forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("%Octa") of the reaction mixture as a function of reaction time in the column. As indicated, the sucrose ester product comprised about 73% octaester and had an I-bar of about 7.61 at 4.75 hours into the reaction conducted in the column.

21. Pages 125-126 of Exhibit 48 describe a sucrose polyester reaction which I conducted on May 7 and 8, 1990 and which was intended to further evaluate the use of the packed column reactor during a later stage of the sucrose polyester reaction. As described at pages 125-126, the top of the packed column reactor was operated at a temperature of about 273-275°F and a pressure which was initially set at about 150 mm Hg and then adjusted to about 50-52 mm Hg, while the bottom of the column was operated at a temperature of about 103-109°C and a pressure of about 70-73 mm Hg. Page 126 sets forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") as a function of time in the column.

22. Pages 128-133 of Exhibit 48 describe three sucrose polyester reactions which I conducted on May 22, 23 and 24, 1990 and which were intended to evaluate the use of a fifteen tray column reactor during a later stage of the sucrose polyester reaction. As described at lines 1-4 of page 128, a goal of these experiments was to achieve 75% octaester-containing product using the fifteen tray column reactor. As described at page 130, lines 19 and 30, page 132, lines 18, 25 and 32, and page 133, line 2, the top of the tray column reactor was operated at pressures of about 32-38 mm Hg, while the bottom of the column was operated at pressures of about 76-95 mm Hg. Pages

131 and 133 set forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") as a function of time in the column.

23. Pages 134-135 of Exhibit 48 describe a sucrose polyester reaction which I conducted on May 31, 1990 and which was intended to further evaluate the use of the fifteen tray column reactor during a later stage of the sucrose polyester reaction. As described at page 134, line 16-page 135, line 9, the top of the column was operated at a pressure of about 34 mm Hg and a temperature of from about 270°F to about 273°F while the bottom of the column was operated at a pressure of about 82 mm Hg and a temperature of from about 188°F to about 205°F. Lines 18-26 of page 135 set forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") as a function of time. As indicated, the sucrose ester product comprised about 99% octaester and had an average degree of esterification of about 7.98 at 3.25 hours into the reaction conducted in the column.

24. Pages 136-139 of Exhibit 48 describe two sucrose polyester reactions which I conducted on June 1 and 4, 1990 and which were intended to further evaluate the use of the fifteen tray column reactor during a later stage of the sucrose polyester reaction. As described at page 136, lines 22-27, and page 138, line 20-page 139, line 6, the top of the tray column reactor was operated at pressures of about 34-35 mm Hg and temperatures of about 270°-273°F, while the bottom of the column was operated at pressures of about 84-87 mm Hg and temperatures of about 199°-204°F. Pages 137 and 139 set forth the I-bar ("$\bar{I}$") of the sucrose ester product and the percent of total sucrose esters as octaester ("% Octa") as a function of time in the column.

25. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the present application or any patent issued thereon.

Respectfully submitted,

By: _____
Steven R. Alexander

Date: 4/8/99

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